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AN ELECTRON DIFFRACTION CAMERA OF SIMPLIFIED DESIGN

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Plate IX A & B

ABSTRACT. A simple electron diffraction camera has been designed where the majority of the specimen motions required for all types of diffraction work has been transmitted into vacuum through a Wilson seal using a rubber gasket only. Diffraction photographs can be taken from four specimens without disturbing the vacuum and with an accuracy better than 1%. The camera is adapted to the electron microscope after its projector lens which can be adjusted to have a shadow micrograph of the specimen under examination.

INTRODUCTION

For electron diffraction work, various rotational and translational motions have to be imparted to the specimen while inside the vacuum The transmission of these motions is generally effected with the help of sylphon bellows and ground joints (Finch and Quarrel, 1933, Germer, 1935; Thiessen and Schoon, 1937, Pinsker, 1953). The conical ground joints require frequent greasing for the maintenance of proper vacuum with the possibility of the contamination of the specimen (Hillier and Baker, 1946). As electron diffraction work requires extreme purity of the specimen, it is preferable to avoid the greased ground joints. Moreover, for transmission of all possible types of motion, the specimen holding mechanism may often be very complicated. In the present work, a specimen chamber is designed in a very simple way avoiding the ground joints

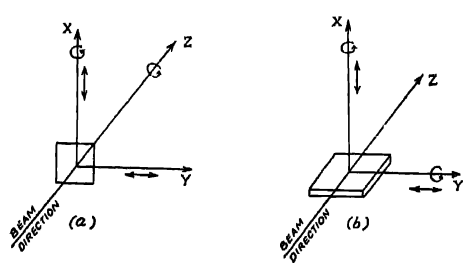

Fig. 1. a) The specimen motions required for transmission diffraction.
b) The specimen motions required for reflection diffraction.

and sylphon bellows. Here the necessary motions have been imparted through a particular type of Wilson seal (Cailson, 1941) using only rubber gaskets.

DESCRIPTION

The different specimen motions required are illustrated in the figures 1(a) and (b). Figure 1(a) refers to the transmission electron diffraction. With the electron beam travelling in the positive direction of the Z-axis, the specimen region giving diffraction patterns of clearest and sharpest definition is to be selected first. This requires a provision for the motion of the specimen along X- and Y-axes. To determine the orientation of the crystals the specimen must be rotated about two axes: the X- axis in order to obtain a different inclination of the crystal planes towards the electron beam and the Z-axis in order to change the orientation in azimuth (important for the study of monocrystalline films). Figure 1(b) refers to the reflection electron diffraction. In this case the correct position is obtained by turning it about the Y- axis and by displacing it either side in the direction of the X-axis. A motion along the Y- axis will leave scope for exploring the specimen. Finally in the case of the single crystal one must be able to rotate the specimen about the X-axis.

In the present work, the realisation of the majority of these motions was made by the use of a Wilson seal of the type shown in figure 2. From figure 2 it

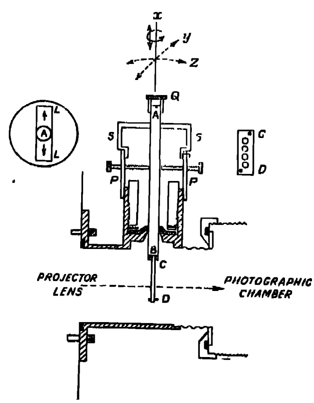

Fig. 2. The sectional diagram of the specimen chamber.

will be evident that the specimen has got both rotational and translation motion along X-axis. Translation is also possible along Y- and Z- axes but they are not linear. It can be easily seen that this will not affect in any way the main objectives since the motions required are very small. The only specimen movements that are not permissible are (a) rotational motion along Y- axis and (b) rotationl motion along Z-axis.

Figure 2 shows the design of the specimen chamber using the above type of Wilson seal. The specimens for transmission work are held inside the grooves in a rectangular brass plate CD, drawn out from one end of the brass rod AB. The specimens are held on the collodion covered steel wire mesh normally used for the electron microscopy. To keep the specimens fixed in position during observation, copper rings are placed over these wire meshes and kept tight by means of another thin brass plate screwed into the former one. Four specimens can be examined one after another and within a very short time without disturbing the experimental conditions. To impart motions to the specimen, four screws at right angles to each other and all in the same horizontal plane pass through a brass cylinder, PP. The rotatory motion is imparted by means of the brass knob Q. Another brass cylinder SS, tightly fitted to PP and fixed in position by a screw, bears a rectangular slot LL on the top of width exactly equal to the diameter of the brass rod AB. The rectangular slot can be fixed normal to the electron optical axis of the camera and thus the motion of the specimen rod is restricted perpendicular to the beam. This prevents any variation of the diffraction during the examination of one specimen or in the course of changing from one length specimen to the next.

The specimen chamber is adapted to the horizontal electron microscope (Das Gupta *et al.*, 1948) built in this laboratory after its projector stage (Hillier *et al*, 1942; Cowley, 1953). There is no magnetic element between the diffraction specimen and the sensitive screen and hence there is no chance of any distortion of the diffraction diagram. Further, a large diffraction angle and hence greater number of diffraction rings are obtained for this position of the specimen.

Figure 3 shows the electron optical ray diagram for diffraction. The condenser lens, C forms an image of the cross over X from the electron gun. The

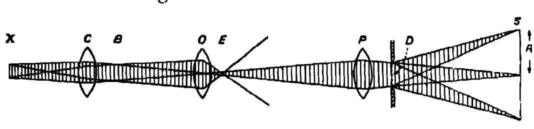

Fig. 3. Electron optical ray diagram for the diffraction work.

position of this image can be varied by varying the condenser current. The smallest cross section B of the beam from the condenser lens is focussed by the objective, O in front of the projector lens P at E. This is again focussed by the projector lens on the final screen in order to obtain sharp diffraction rings. The specimen is placed in the position D. The intensity of the diffraction diagram can be controlled by adjusting the objective and the condenser currents. The projector lens current can also be adjusted to obtain an electron optical shadow micrograph of the specimen under observation. This allows an examination of the nature and the distribution of the diffraction specimen at a magnification of about 30X and ensures proper selection of the specimen area for diffraction work.

RESULTS

Plate IX A shows the diffraction photographs of the four gold films of the same thickness placed at the four positions of the holder. The ring diameters of the individual photographs were measured and are given in Table 1. The maximum deviations in the measured diameters of the corresponding rings as percentage of the mean diameter are given in the last column of the table. An accuracy better than 1% is achieved with this system.

TABLE I

Measurements on the diffraction patterns from the four gold films

hkl	Measured diameters (cm) from Plate IXA				Maximum deviation
	(a)	(b)	(c)	(d)	
111	1.462	1.465	1.467	1.460	0.5%
200	1.699	1.698	1.692	1.686	0.9%
220	2.389	2.380	2.389	2.382	0.4%
113	2.797	2.792	2.780	2.780	0.6%

The calibration of the camera was made from the diffraction pattern of a thallium chloride specimen (Plate IXB). $L\lambda$ was found to be 1.8464×10^{-8} cm^2. The normal diffraction pattern, the oblique texture pattern and shadow micrograph ($125\times$) of a CdI_2 specimen are shown in Plate IXB b, c and d respectively.

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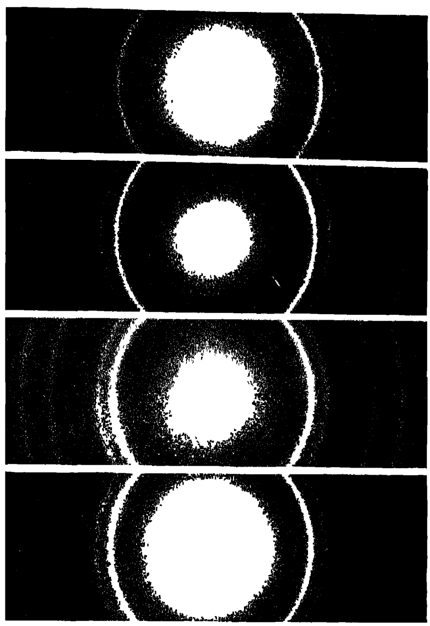
PLATE IX A

(a)

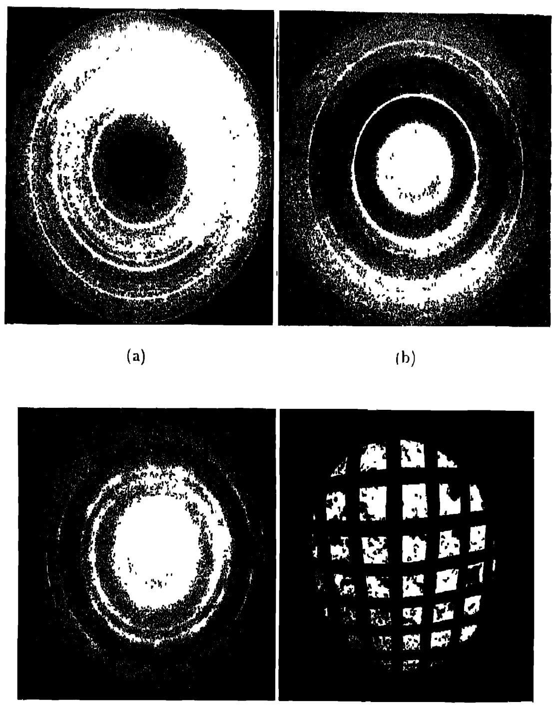
(b)

(c)

(d)



Electron diffraction photographs taken successively from four gold films of the same thickness from the four positions of the holder.



(a) (b)

(c) (d)

Electron diffraction photographs.

(a) Electron diffraction pattern of thallium chloride

(b) Normal diffraction pattern of CdI_2

(c) Oblique texture pattern of CdI_2

(d) Electron shadow micrograph of CdI_2 (125×).